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THE DISSOLUTION KINETICS OF LITHIATED NiO IN AQUEOUS
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**13. ABSTRACT**

The factors controlling the dissolution of metal oxides are of major importance in understanding the role of such oxides in the control of corrosion through the formation of passivation films and the use of various oxides as battery cathodes. The dissolution kinetics of mosaic lithiated nickel oxide have been examined in concentrated $H_2SO_4$ and HCl solutions as a function of potential and found to be strongly dependent on potential with the rate increasing with increasingly anodic potentials and passing through a maximum in the $H_2SO_4$ solutions. This behavior corresponds to control of the dissolution kinetics by transmission of the cations over a potential energy barrier at potentials cathodic to the potential for the dissolution maximum and by anions over such a barrier at potentials anodic to the maximum. The temperature dependence indicates Arrhenius behavior with an enthalpy of activation of $\sim 23$ kcal/mole. Complexing agents such as EDTA have been found to increase the dissolution rate. At relatively anodic potentials, $O^{18}$ labelling experiments indicate that the generation of $O_2$ involves to a significant extent lattice $O^=$, probably through a mechanism involving the p-carriers falling into $O^=$ surface states.

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TECHNICAL REPORT NO. 40

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Case Western Reserve University
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# THE DISSOLUTION KINETICS OF LITHIATED NiO IN AQUEOUS ACID SOLUTIONS

by

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INTRO: ..ION

Transition metal oxides are of major importance in a number of areas of electrochemistry, foremost of which are the corrosion of transition metals and their alloys, various battery systems, and electrochemical catalysis. A study of the mechanism of oxide dissolution and the factors which control the dissolution kinetics should lead to a better understanding of the role of oxides in these various areas of electrochemistry.

Previous work on the study of oxide dissolution mechanisms have been reviewed by Diggle (1). Various workers have found that the dissolution rates are usually potential dependent. The theoretical interpretation of the potential dependence of the dissolution rate of metal oxides has been considered by Engell (2) and Vermilyea (3). These treatments consider the dissolution of ionic oxides when the process is to be controlled by the transfer of ions across the oxide-electrolyte interface.

The rate of dissolution of an ionic crystal with the back reaction negligible is given by Vermilyea (3) as follows:

$$r = \frac{2r_f}{\exp - \dfrac{\alpha_+ z_+ F\eta}{RT} + \exp - \dfrac{\alpha_- z_- F\eta}{RT}} \tag{1}$$

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where $\eta = \phi_o - \phi_{o,f} = E - E_f$; $\phi_o$ and $\phi_{o,f}$ are the potential drops between oxide surface and outer Helmholtz plane in the solution corresponding to electrode potentials $E$ and $E_f$; $E_f$ is the freely dissolving potential, $r_f$ is the dissolution rate corresponding to $\eta = 0$; $\alpha$ and $z$ is the transfer coefficient and the charge number (including sign) of cation (+) and anion (-). This equation is applicable to oxides such as lithiated NiO if there are no complications with either space charge or preferential site dissolution.

Preliminary studies of the dissolution properties of mosaic crystal lithiated NiO have been carried out at CWRU by Yohe et al. (4). These studies indicated a pronounced potential dependence of the dissolution rate in acid solutions. The space charge properties of the NiO(Li) - electrolyte interface have been examined by Tench and Yeager (5,6) who report an effective flat-band potential ($E_{fb}$) of ~1.1V vs SHE in acid solution. At potentials anodic to $E_{fb}$, the majority of the potential drop is across the Helmholtz plane and the treatment of Vermilyea (3) may be applicable. At potentials cathodic to $E_{fb}$, most of the potential drop is across a space charge region within the semiconducting oxide and eq. 1 would not be applicable.

The present study of lithiated NiO has involved the examination of 1) the dependence of dissolution on the electrode potential and the pH of the solution and 2) the relationship between dissolution and the simultaneous residual currents.

EXPERIMENTAL PROCEDURES

Electrodes were prepared from <100> mosaic crystals of NiO(Li) which were grown and doped as described earlier (5). The dissolution experiments were carried out at 65 - 95°C (mostly at 95°C) in an all Pyrex cell under purified helium gas atmosphere in 1 $\underline{N}$ HCl and 1 $\underline{N}$ $H_2SO_4$ under potentiostatic conditions. The solutions were prepared from redistilled

reagent grade solutions with triple-distilled water followed by a treatment
of purified activated carbon. The counter (Pt) and reference (Ag/AgCl)
electrodes were isolated from the working electrode compartment by the
use of salt-bridges of the same solution. Precautions were taken to
minimize contamination of the working electrode. A Wenking potentiostat
was used for the control of potential. The residual currents were monitored
with an electrometer (Hewlett-Packard 419A DC null voltmeter). The dissolu-
tion rates were evaluated by analyzing the solutions for $Ni^{2+}$ with a spectro-
photometer using DMG as a complexing agent (7) [sensitive to trace $Ni^{2+}$ down
to 0.005 ppm]. The reproducibility of the dissolution results was $\pm$ 5%
or better.

RESULTS AND DISCUSSION

1. Dependence of Dissolution on Electrode Potential

The dissolution rate data (Fig. 1) for $H_2SO_4$ indicate a maximum in
the rate at anodic potentials with a Tafel slope on either side of 0.12
to 0.14 V/decade. This is in accord with eq. 1; i.e., below the maximum,
the rate is controlled by the barrier for cation transfer and above the
maximum by that for anion transfer. The charges of the species being
transferred are uncertain but if $z_+$ is +2 and $z_-$ is -1, then for the
$H_2SO_4$, $\alpha_+ = 0.31$ and $\alpha_- = 0.62$. The maximum could not be observed in HCl
because of $Cl_2$ evolution but the Tafel slope of ∿0.16 V/decade corresponds
to $\alpha_+ = 0.23$.

The change in the Tafel slope in the region 1.2 to 1.3V is probably
a consequence of a large change in the $Ni^{3+}/Ni^{2+}$ ratio on the surface,
according to the reaction $2 NiO + H_2O \rightarrow Ni_2O_3 + 2 H^+ + 2e$. Only in the
potential region below 0.8 $V^a$ is the dissolution rate dependent on the Li

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[a] All potentials are expressed vs the standard hydrogen electrode (SHE).

concentration in the oxide.[b] This region has been found to correspond to
exhaustive depletion in earlier studies (5) of the space charge properties
of such electrodes. At more anodic potentials the change in potential drop
across the space charge region is small.

## 2. Dependence of Dissolution on the pH of the Solution

The effect of pH on the dissolution rate (r) was studied at 95°C
in HCl + KCl with the total ionic strength held at unity (Fig. 2). The
log r decreased linearly with pH, and $(\partial\log r/\partial pH)_E$ = -0.56, -0.53, and
-0.32 at E = 0.9, 1.1, and 1.3V, respectively.

Theoretical prediction of $(\partial\log r/\partial pH)_E$ can be made by introducing
the relationship between the potential $\phi_o$ and $H^+$ activity at constant
applied potential, i.e.,

$$\phi_o = \frac{RT}{F} \ln \frac{a_H^+}{(a_H^+)_o} + \text{constant} \tag{2}$$

with cation transfer rate controlling and negligible back reaction

$$r = \vec{r}_+ = n_+\vec{k}_+ \exp \frac{\alpha_+ z_+ F\phi_o}{RT} \tag{3}$$

Combination of eqs. 2 and 3 yields

$$(\partial\log r/\partial pH)_E = -\alpha_+ z_+ \tag{4}$$

The predicted slope according to eq. 4 using the values $\alpha_+$ = 0.23,
$z_+$ = 2 evaluated from Fig. 1 is -0.46 as compared with an experimental
value of $\sim$ -0.5. Thus the agreement between experimental results and
theory is satisfactory.

## 3. Effect of Complexing Agent and Redox Couples

The complexing agent (EDTA) was found to increase the dissolution
rate linearly with concentration at 0.765 to 1.165 V (Fig. 3). EDTA
complexes with $Ni^{2+}$. Thus the enhancement of dissolution by EDTA

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[b] The concentrations listed in the legend of this and subsequent figures
were determined from the slopes of the Mott-Schottky plots of the
capacity-potential data (see ref. 5).

provides further evidence that the cation transfer is rate controlling
at potentials cathodic to the maximum in Fig. 1

The introduction of $1.0 \times 10^{-4} \underline{M} Tl^+$ ions into the $1\underline{N}$ HCl solution
had no detectable effect on the dissolution rate. The $E^\circ$ of the $Tl^+/Tl^{3+}$
couple is 1.25 V and hence $Tl^+$ should be oxidized to $Tl^{3+}$ at the more
anodic potentials. If some types of electronic charge transfer
process were rate controlling for the dissolution of the NiO, then
an interaction between this electronic charge transfer step and

the oxidation of the $Tl^+$ might have been expected. Furthermore, if a
large portion of the potential were across the space charge layer
within the oxide, the presence of such redox couple would have influenced
the value for $\phi_o$ at a given applied potential E, and hence, would have
changed the dissolution rate. In the potential range 1.265 V to 1.465 V
where the effect of the $Tl^+$ ion were examined, however, the potential
drop across the diffuse portion of the space charge region in the oxide
is believed to be small (5) and the value for $\phi_o$ is controlled principally
by surface states at a given value of E. Therefore, the lack of any
appreciable effect with the introduction of $Tl^+$ is compatible with the
present interpretation of the dissolution kinetics as under ion transfer
control.

## 4. Residual Currents Attending the Dissolution

Although the residual currents during dissolution were not very
reproducible, small cathodic currents ($10^{-8}$ to $10^{-7}$ A/cm$^2$) were evident
at potentials cathodic to $\sim$0.8 V  (see Fig. 4). The scatter of
the data, however, prevented recognizing any consistent trends with
respect to potential in the range 0.3 to 0.8 V  or lithium contents of the

NiO(Li) electrodes in the range 0.1 to 1.0 cation % Li. For comparison
purposes, the current values equivalent to 1% of the dissolution rates
are also shown in Fig. 4, assuming one electron/nickel ion.

The source of the scatter in the cathodic residual current data
is not clear at this time but may have resulted from impurity effects,
despite the purifications procedures used to safeguard against such
effects in this work (9). A cathodic current is expected to attend
the dissolution because the $Ni^{3+}$ produced by the introduction of Li
into the lattice must be finally converted to $Ni^{2+}$ in the solution
phase. This may proceed through the reduction of $Ni^{3+}$ to $Ni^{2+}$ on the
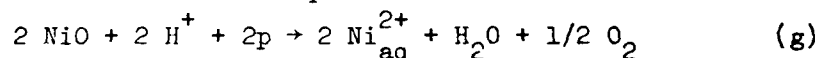NiO(Li) surface preceeding the transfer of $Ni^{2+}$ ions into solution; _i.e._,

$$Ni^{3+}_{surface} \longrightarrow Ni^{2+}_{surface} + p \qquad \text{(a)}$$
$$\downarrow$$
$$Ni^{2+}_{aq}$$
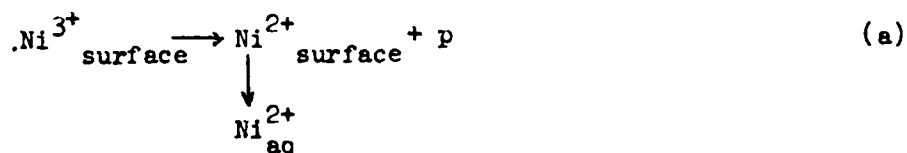
An alternative is the transfer of $Ni^{3+}$ into solution followed by
reduction; _i.e._,

$$Ni^{3+}_{surface} \qquad \text{(b)}$$
$$\downarrow$$
$$Ni^{3+}_{aq} \longrightarrow Ni^{2+} + p$$
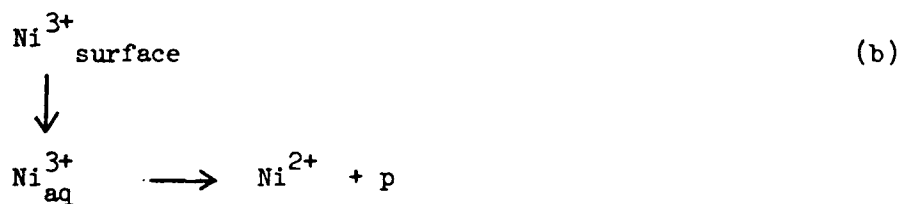
This process seems less likely, however, on the basis of thermodynamic
considerations. The potential of the $Ni^{2+}/Ni^{3+}$ couple for any reasonable
ratio of $Ni^{3+}$ to $Ni^{2+}$ in solution would be very anodic to the voltage
range where the cathodic residual currents are observed.

At potentials anodic to ∿1.0 V an anodic current was
usually observed (see Fig. 4). At potentials more anodic than 1.5 V

the current rapidly increases as $O_2$ is evolved at increasing rates in the $H_2CO_4$ electrolyte (or $Cl_2$ in the HCl). The anodic currents at potentials even well b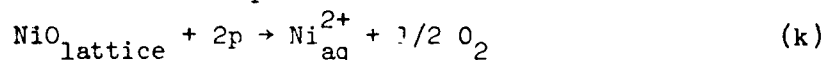elow those of the reversible $O_2$ electrode (1.2 V however, may also still be associated with $O_2$ formation since the anodic formation of $O_2$ from lattice $O^{2-}$ can proceed at potentials less than those required for $O_2$ discharge from water. This is

evident if one considers the coupling of the dissolution process to the formation of the $O_2$ from lattice $O^{2-}$; _i.e._,

$$2\ O^{2-} + 2p \rightarrow 2\ O^- \tag{c}$$

$$2\ O^- + 2\ H^+ \rightarrow 2\ OH \tag{d}$$

$$OH + OH \rightarrow H_2O + 1/2\ O_2 \tag{e}$$

$$2\ Ni^{2+}_{lattice} \rightarrow 2\ Ni^{2+}_{aq} \tag{f}$$

$$2\ NiO + 2\ H^+ + 2p \rightarrow 2\ Ni^{2+}_{aq} + H_2O + 1/2\ O_2 \tag{g}$$
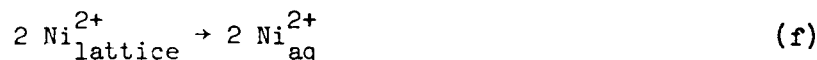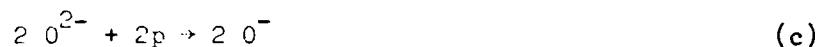
Reactions c and d may be a single step or separate steps as shown. Reaction e undoubtedly involves several component steps. The overall process is given by reaction g, independent of the steps. The standard free energy change from overall process relative to the reaction $2H^+ + 2e \rightarrow H_2$ can be calculated from the standard free energy of formations as listed by Pourbraix (8); NiO, $\Delta G^o_f = -51.3$ kcal/mole; $Ni^{2+}$, $\Delta G^o_f = -11.53$ kcal/mole; $H_2O$, $\Delta G^o_f = -56.5$ kcal/mole. The calculated relative standard free energy change for reaction g is +23.1 kcal/mole corresponding to a standard electrode potential of + 0.5 V.

Alternatively the $O_2$ formation from lattice oxide may involve two p carriers falling into a $O^{2-}$ surface state; i.e.,

$$O^{2-} + 2p \rightarrow O \qquad\qquad\qquad (h)$$

$$O \rightarrow 1/2 \ O_2 \qquad\qquad\qquad (i)$$

$$Ni^{2+}_{lattice} \rightarrow Ni^{2+}_{aq} \qquad\qquad\qquad (j)$$

$$NiO_{lattice} + 2p \rightarrow Ni^{2+}_{aq} + 1/2 \ O_2 \qquad\qquad\qquad (k)$$
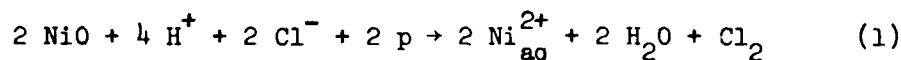
where reactions h and i are multiple step processes. For the overall process k, $\Delta G^\circ = + 39.8$ kcal/mole and $E^\circ = 0.87$ V. Both of these potentials are cathodic to that for discharge of $O_2$ from water. Since the activity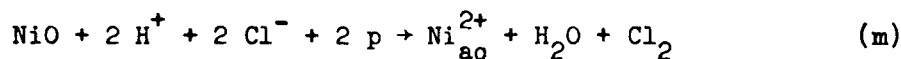 of $Ni^{2+}_{aq}$ is far less than the unit value, the equilibrium potentials may be considerably less anodic than the standard values.

$O^{18}$ - labelling experiments (9) have indicated that the $O_2$ generated on NiO(Li) anodes at more anodic potentials (>1.8 V vs SHE) is produced to a significant extent directly from lattice $O^=$ and not just $H_2O$ of the solution. Unfortunately the limitation of the analytical method prevented the $O^{18}$-labelling experiment from being carried out at less anodic potentials.

In chloride solution, the $O^-$ ions or O atoms would react with $Cl^-$ to form $Cl_2$. The overall reaction in this case would be

$$2 \ NiO + 4 \ H^+ + 2 \ Cl^- + 2 \ p \rightarrow 2 \ Ni^{2+}_{aq} + 2 \ H_2O + Cl_2 \qquad (l)$$

with $\Delta G^\circ = +29.2$ kcal/mole; $E^\circ = 0.63$ V, and

$$NiO + 2 \ H^+ + 2 \ Cl^- + 2 \ p \rightarrow Ni^{2+}_{aq} + H_2O + Cl_2 \qquad (m)$$

with $\Delta G^\circ = +46$ kcal/mole; $E^\circ = 1.0$ V

These voltages are also much less than the standard values for the anodic formation of $Cl_2$ from $Cl^-$.
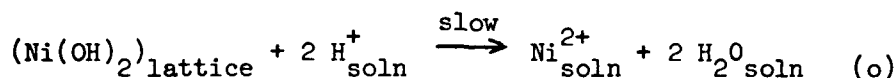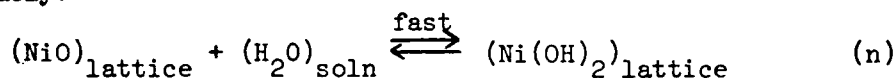
## 5. Temperature Dependence

Figure 5 represents the Arrhenius plot of the observed dissolution rate vs $1/T$ at $E = 1.0$ and $1.4$ V. These two potentials fall in the regions of high and low Tafel slope. The apparent activation energies may be evaluated from the slopes with the equation

$$(\Delta H^{\ddagger})_E = -2.303 \ R \ [\frac{\partial \log r}{\partial (1/T)}]_E \tag{5}$$
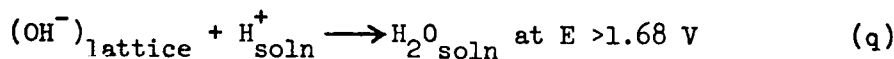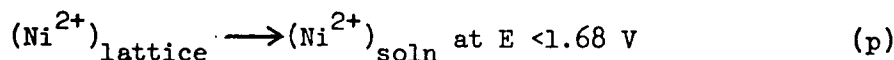
and are 22.9 kcal/mole at 1.0 V and 20.8 kcal/mole at 1.4 V. Thus the potential dependence of $\Delta H^{\ddagger}$ is relatively small. The potential at which $\Delta H^{\ddagger}$ would be most interesting is that corresponding to $\phi_o = 0$, since at this potential the $Ni^{2+}$ would not be influenced by any field between the Helmholtz planes in the oxide and that in the solution. Unfortunately the potential corresponding to $\phi_o = 0$ is unknown but probably is not more than a few tenths of a volt from the flat band potential $E_{fb}$ even with interference from anomalous surface states. At 25°C, $E_{fb} = \sim 1.1$ V and even at the considerably higher temperatures involved in Fig. 5, it would still be expected to be rather close to this value. Therefore, the value of $\Delta H^{\ddagger}$ at $\phi_o = 0$ should be close to the observed value of 23 kcal/mole at 1.0 V. This value is an order of magnitude smaller than the total energy of interaction of the $Ni^{2+}$ with the six inner coordination sphere ligands in either the solid oxide phase or the solution phase. Assuming $Ni^{2+}$ transfer across the interface as rate controlling, this implies that in the activated complex most of the ligand interactions of the inner coordination sphere are still intact.

## 6. Proposed Dissolution Mechanism

The dissolution kinetics for NiO(Li) do exhibit the type of potential and pH behavior expected from the Vermilyea treatment (3) for ion transfer as rate controlling. It is not possible to identify the nature of the species being transferred, however, in various potential regimes from the observed Tafel slopes because of uncertainty as to the transfer coefficient $\alpha$. The authors suggest that the following mechanism explains the observed results quite well although not necessarily uniquely.

$$(NiO)_{lattice} + (H_2O)_{soln} \underset{}{\overset{fast}{\rightleftharpoons}} (Ni(OH)_2)_{lattice} \qquad (n)$$

$$(Ni(OH)_2)_{lattice} + 2 H^+_{soln} \xrightarrow{slow} Ni^{2+}_{soln} + 2 H_2O_{soln} \qquad (o)$$

with either one of the following steps as rate controlling:

$$(Ni^{2+})_{lattice} \longrightarrow (Ni^{2+})_{soln} \text{ at } E < 1.68 \text{ V} \qquad (p)$$

$$(OH^-)_{lattice} + H^+_{soln} \longrightarrow H_2O_{soln} \text{ at } E > 1.68 \text{ V} \qquad (q)$$

This discussion has not considered the morphology of the <100> NiO(Li) surface during the dissolution nor is such adequately represented in eq. 1. For the fully quantitative interpretation of such kinetic studies, the morphological aspects of the surface dissolution must be taken into account. Earlier work (10) has shown that at potentials cathodic to +0.8 V the surface is uniformly attacked with no etch pits evident using _in situ_ optical microscopic examination of magnifications up to 800X during the dissolution even over periods of up to 7 hr. At potentials anodic to +0.9 V in 1 N HCl and +1.0 V in 1 N $H_2SO_4$, however, etch pits were clearly evident even after the first few minutes. This difference in behavior above and below potentials

of ~0.9 V is not just a matter of the slow dissolution rate at less

anodic potentials making it more difficult to show up etch pits.

Acknowledgement:  The authors acknowledge the support of the research

by the U.S. Office of Naval Research.

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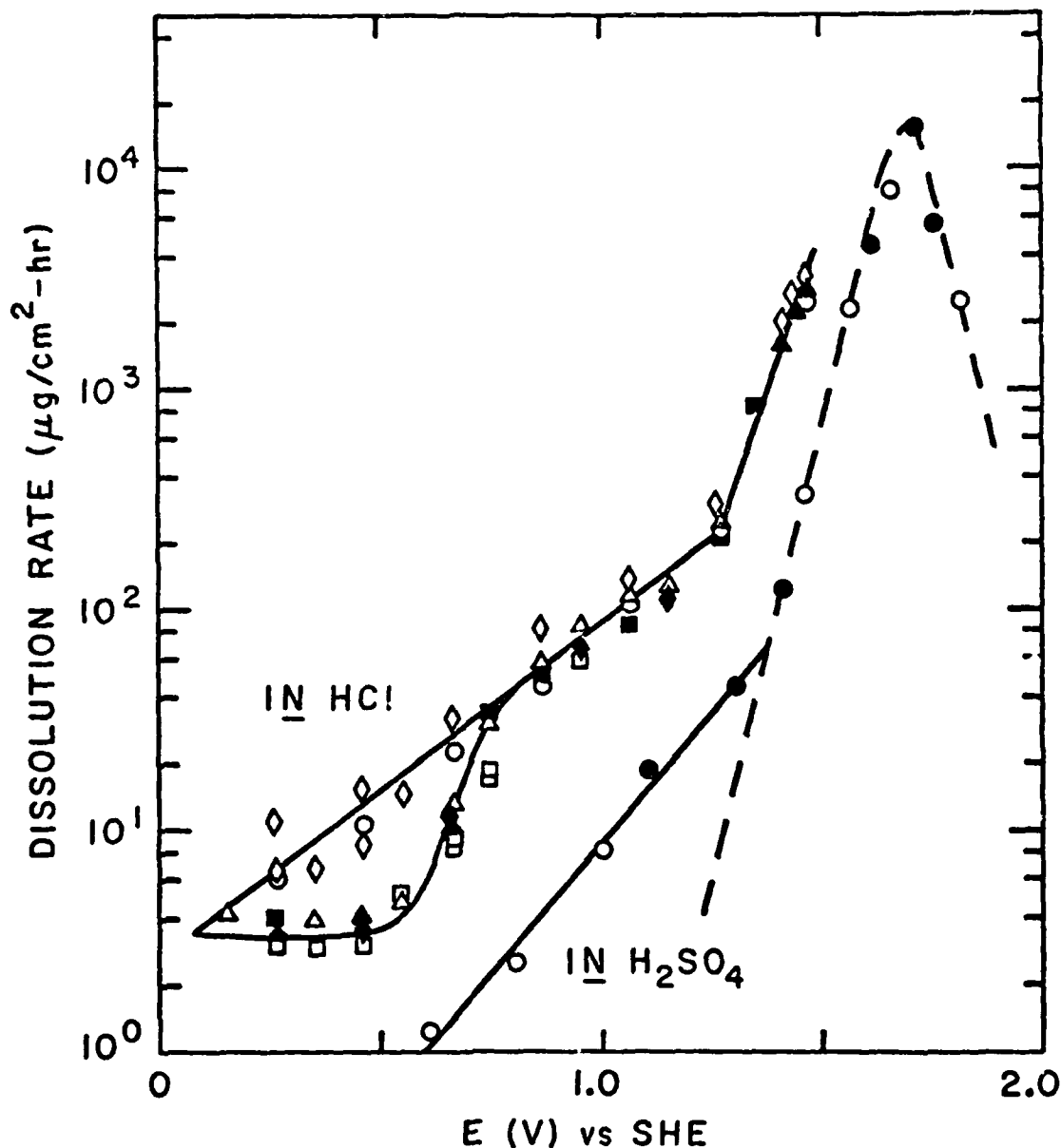    tion Properties of Nickel Oxide", Tech. Report 2, ibid., 1966.

Figure 1. Dissolution of lithiated nickel oxide in helium saturated 1 $\underline{N}$ HCl and 1 $\underline{N}$ $H_2SO_4$ solution at 95°C. Symbols corresponding to different group of electrodes:

◊ ● Group 1, $(C_{Li})_{MS}$ = 0.88%; ▢ ■ △ Group 3, $(C_{Li})_{MS}$ = 0.26%;

◇ Group 2, $(C_{Li})_{MS}$ = 0.33%; ▲ ◆ Group 4, $(C_{Li})_{MS}$ = 0.12%

Dashed line: Calculated from eq. 1 with the following parameters: $\alpha_+ z_+$ = 0.62, $\alpha_- z_-$ = -0.62, $r_f$ = 15,000 µg/cm²-hr, $\phi_f$ = 1.68 V $\underline{vs}$ SHE. [Concentrations of Li in cation -%, based on the slopes of the Mott-Schottky plots.]
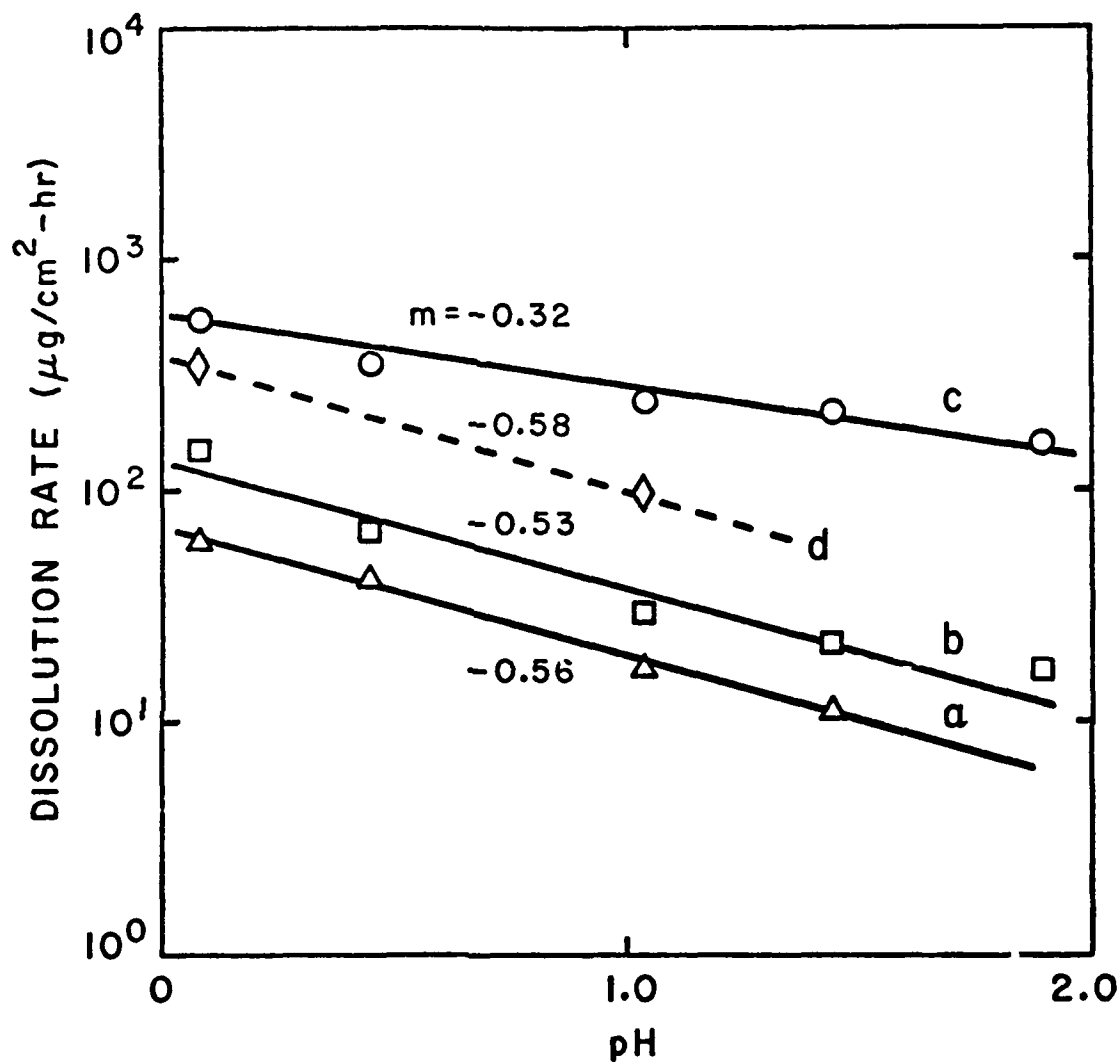
Figure 2.  Dissolution of lithiated NiO $[(C_{Li})_{MS} = 0.26\%]$
in HCl + KCl solution as a function of pH at various electrode
potentials; a) 0.9 V, b) 1.1 V, c) 1.3 V, d) 1.0 V vs SHE.
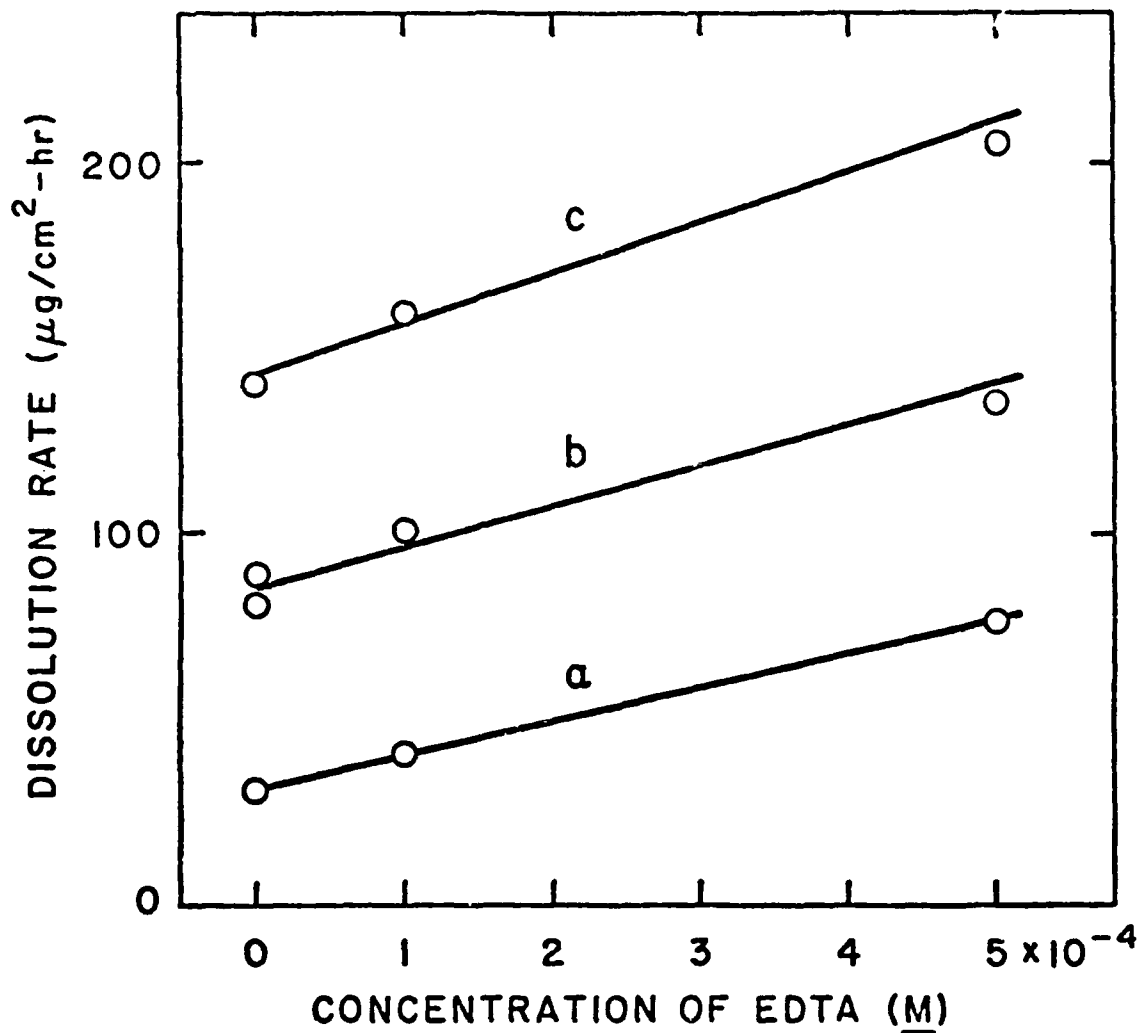Solid lines:  present work, dashed line:  Yohe et al. (4).

Figure 3.  Dissolution of lithiated NiO (0.26% Li) in 1 $\underline{N}$ HCl
solution with and without EDTA present at 95°C and at
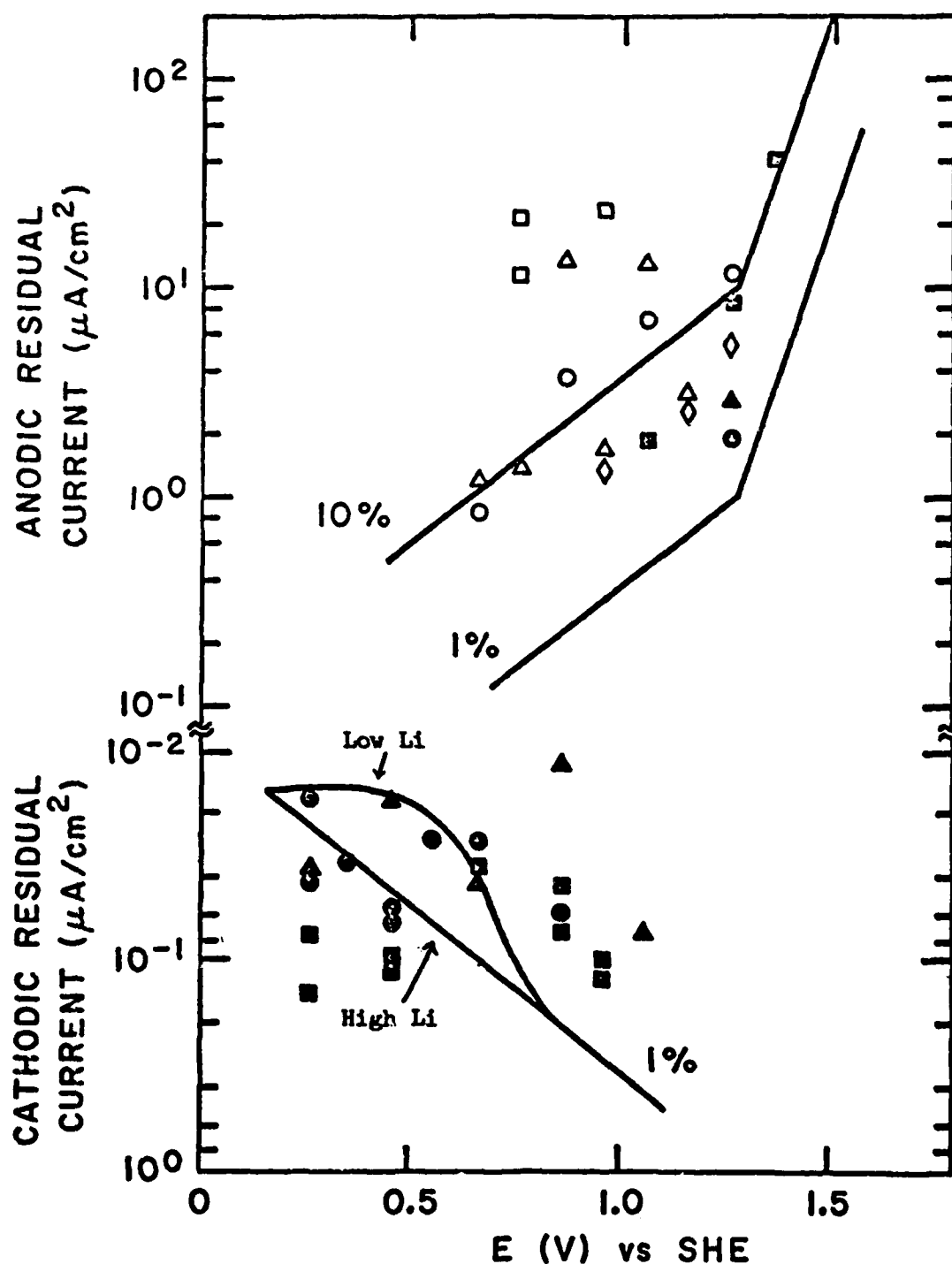potentials: a) 0.765 V,  b) 0.965 V, and c) 1.165 V $\underline{vs}$ SHE.

Figure 4. Residual currents recorded at the end of the
dissolution of (Li)NiO in helium saturated 1N HCl solution
at 95°C. Symbols are the actual data corresponding to
electrodes shown in Figure 1. Solid curves are calculated
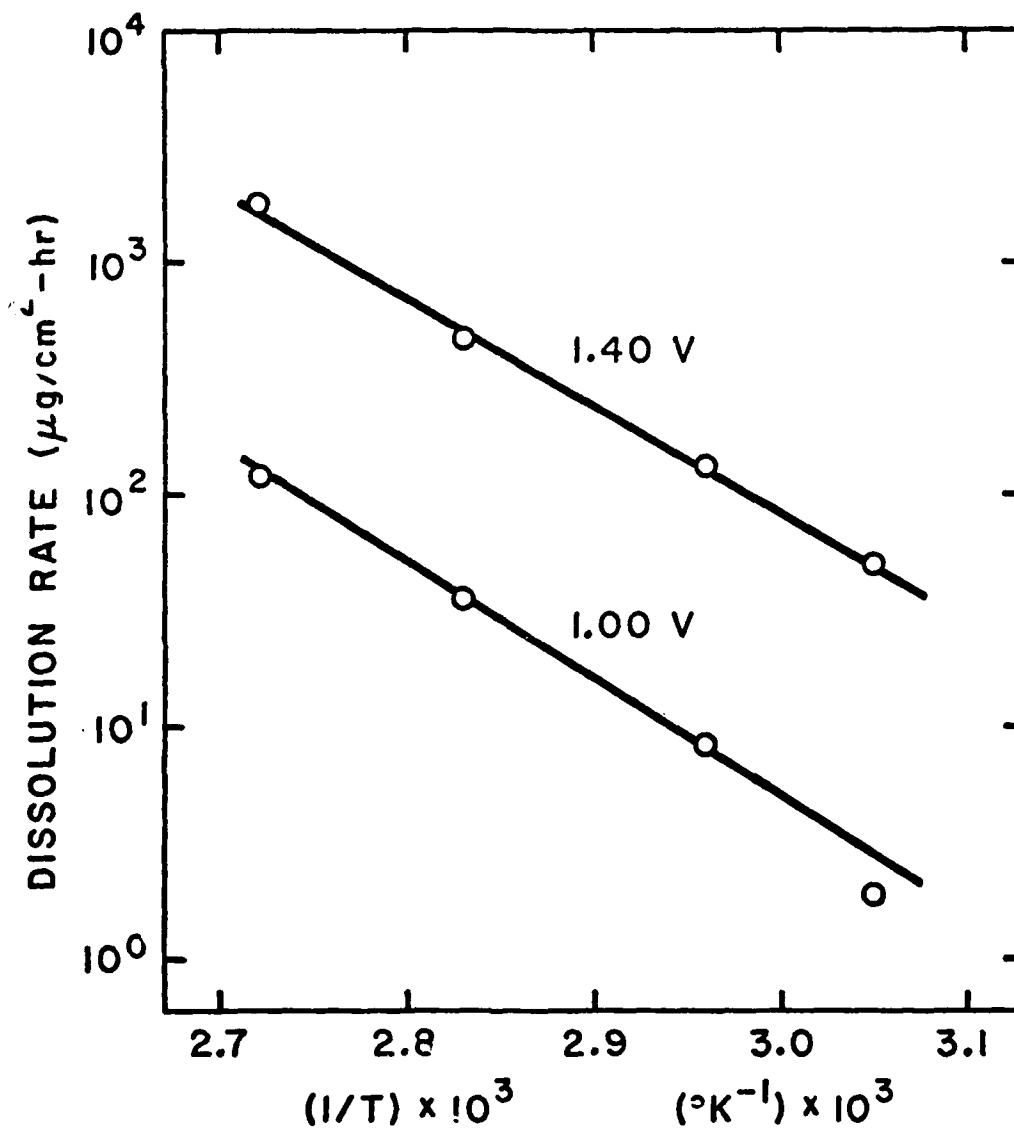currents corresponding to 1 and 10% of dissolution rates.

Figure 5. Dissolution of lithiated NiO in He-gas saturated
1 N HCl solution as a function of temperature at constant
electrode potentials (vs SHE) for 0.26 % Li.